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PTO/SB/05 (4/98)

Approved for use through 9/30/2000. OMB 0651-0032

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UTILITY PATENT APPLICATION TRANSMITTAL

Ily for non provisional applications under 37 C.F.R. § 1.53(b)

Attorney Docket No.	C 2109 COGG
First Inventor or Application Identifier	BUETTGEN, Karl-Heinz
Title	PROCESS FOR THE PRODUCTION OF DEACIDIFIED TRIGLYCERIDES
Express Mail Label No.	EL544993079US

APPLICATION ELEMENTS

MPEP chapter 600 concerning utility patent application contents.

<input checked="" type="checkbox"/> X	* Fee Transmittal Form (a.g., PTO/SB/17) (Submit an original and a duplicate for fee processing)	
<input checked="" type="checkbox"/> X	Specification (Total Pages 9) (preferred arrangement set forth below)	9

- Descriptive title of the Invention
- Cross References to Related Applications
- Statement Regarding Fed sponsored R&D
- Reference to Microfiche Appendix
- Background of the Invention

- Brief Summary of the Invention
- Brief Description of the Drawings (if filed)
- Detailed Description
- Claim(s)
- Abstract of the Disclosure

3. ☐ Drawing(s) (35 U.S.C. 113) [Total Sheets ☐]4. ☒ Oath or Declaration [Total Pages ☐]

- a. ☐ Newly executed (original or copy)
- b. ☐ Copy from a prior application (37 C.F.R. § 1.63(d))
(for continuation/divisional with Box 16 completed)

- i. ☐ **DELETION OF INVENTOR(S)**
Signed statement attached deleting
inventor(s) named in the prior application, see
37 C.F.R. §§ 1.63(d)(2) and 1.33(b).

***NOTE FOR ITEMS 1 & 3: IN ORDER TO BE ENTITLED TO PAY SMALL ENTITY
FEES, A SMALL ENTITY STATEMENT IS REQUIRED (37 C.F.R. § 1.17), EXCEPT
IF ONE FILED IN A PRIOR APPLICATION IS RELIED UPON (37 C.F.R. § 1.28).**

ADDRESS TO: Assistant Commissioner for Patents
Box Patent Application
Washington, DC 20231

5. ☐ Microfiche Computer Program (Appendix)
6. Nucleotide and/or Amino Acid Sequence Submission
(if applicable, all necessary)
- a. ☐ Computer Readable Copy
- b. ☐ Paper Copy (identical to computer copy)
- c. ☐ Statement verifying identity of above copies

ACCOMPANYING APPLICATION PARTS

7. ☐ Assignment Papers (cover sheet & document(s))
8. ☐ 37 C.F.R. § 3.73(b) Statement ☐ Power of Attorney
(when there is an assignee)
9. ☐ English Translation Document (if applicable)
10. ☐ Information Disclosure
Statement (IDS)/PTO-1449 ☐ Copies of IDS Citations
11. ☐ Preliminary Amendment
12. ☒ Return Receipt Postcard (MPEP 503)
(Should be specifically itemized)
13. ☐ Small Entity
Statement(s) ☐ Statement filed in prior application,
Status still proper and desired
(PTO/SB-012)
14. ☒ Certified Copy of Priority Document(s)
(if foreign priority is claimed)
15. ☐ Other: _____

16. IF A CONTINUING APPLICATION, check appropriate box, and supply the requisite information below and in a preliminary amendment:

☐ Continuation ☐ Divisional ☐ Continuation-in-part (CIP)

of prior application No. _____ / _____

Prior application information: Examiner _____

Group / Art Unit: _____

For CONTINUATION or DIVISIONAL APPS only: The entire disclosure of the prior application, from which an oath or declaration is supplied under Box 4b, is considered a part of the disclosure of the accompanying continuation or divisional application and is hereby incorporated by reference. The incorporation can only be relied upon when a portion has been inadvertently omitted from the submitted application parts.


17. CORRESPONDENCE ADDRESS

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23657

or ☒ Customer address below

Name	John E. Drach		
Address			
City	State	Zip Code	
Country	Telephone	610-278-4925	Fax 610-278-6548

Name (Print/Type)	John E. Drach	Registration No. (Attorney/Agent)	32,891
Signature		Date	November 21, 2000

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for FY 2000**

Patent fees are subject to annual revision.
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Otherwise large entity fees must be paid. See Forms PTO/SB/09-12.
See 37 C.F.R. §§ 1.27 and 1.28.

TOTAL AMOUNT OF PAYMENT **(\$)** 710**Complete if Known**

Application Number	
Filing Date	
First Named Inventor	BUETTGEN, Karl-Heinz
Examiner Name	
Group/Art Unit	
Attorney Docket No.	C 2109 COGG

METHOD OF PAYMENT (check one)

- 1.
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- The Commissioner is hereby authorized to charge indicated fees and credit any over payments to:

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FEE CALCULATION

Large Fee Code	Entity Fee Code	Small Fee Code	Entity Fee Code	Fee Description	Fee Paid
101	710	201	345	Utility filing fee	710
106	310	206	155	Design filing fee	
107	480	207	240	Plant filing fee	
108	690	208	345	Reissue filing fee	
114	150	214	75	Provisional filing fee	

SUBTOTAL (1) **(\$)** 710**2. EXTRA CLAIM FEES**

Extra Claims	Fee from below	Fee Paid
Total Claims 10 -20**= 0 X \$18.00 = 0		
Independent Claims 1 -3**= 0 X \$18.00 = 0		
Multiple Dependent 1	\$260.00	0

**or number previously paid, if greater; For Reissues, see below

Large Fee Code	Entity Fee Code	Small Fee Code	Entity Fee Code	Fee Description
103	18	203	9	Claims in excess of 20
102	78	202	39	Independent claims in excess of 3
104	260	204	130	Multiple dependent claim, if not paid
109	78	209	39	** Reissue independent claims over original patent
110	18	210	9	** Reissue claims in excess of 20 and over original patent

SUBTOTAL (2) **(\$)** .00**FEE CALCULATION (continued)****3. ADDITIONAL FEES**

Large Fee Code	Entity Fee Code	Small Fee Code	Entity Fee Code	Fee Description	Fee Paid
105	130	205	85	Surcharge - late filing fee or oath	
127	50	227	25	Surcharge - late provisional filing fee or cover sheet	
139	130	139	130	Non-English specification	
147	2,520	147	2,520	For filing a request for reexamination	
112	920*	112	920*	Requesting publication of SIR prior to Examiner action	
113	1,840*	113	1,840*	Requesting publication of SIR after Examiner action	
115	110	215	55	Extension for reply within first month	
116	380	216	190	Extension for reply within second month	
117	870	217	435	Extension for reply within third month	
118	1,360	218	680	Extension for reply within fourth month	
128	1,850	228	925	Extension for reply within fifth month	
119	300	219	150	Notice of Appeal	
120	300	220	150	Filing a brief in support of an appeal	
121	260	221	130	Request for oral hearing	
138	1,510	138	1,510	Petition to institute a public use proceeding	
140	110	240	55	Petition to revive - unavoidable	
141	1,210	241	605	Petition to revive - unintentional	
142	1,210	242	605	Utility issue fee (or reissue)	
143	430	243	215	Design issue fee	
144	580	244	290	Plant issue fee	
122	130	122	130	Petitions to the Commissioner	
123	50	123	50	Petitions related to provisional applications	
126	240	126	240	Submission of Information Disclosure Sheet	
581	40	581	40	Recording each patent assignment per property (times number of properties)	
146	690	246	345	Filing a submission after final rejection (37 C.F.R. § 1.129(a))	
149	690	249	345	For each additional invention to be examined (37 C.F.R. § 1.129(b))	

Other fee (specify) _____

Other fee (specify) _____

* Reduced by Basic Filing Fee Paid

SUBTOTAL (3) **(\$)** .00**SUBMITTED BY**Name (Print/Type) **John E. Drach**Registration No. **32,891**
(Attorney/Agent)**Complete (if applicable)**Telephone **(610) 278-4925**

Signature

Date

November 21, 2000

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Number EL544993079US.**

TITLE OF THE INVENTION

Process for the Production of Deacidified Triglycerides

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CROSS-REFERENCE TO RELATED APPLICATIONS

Not applicable.

10

STATEMENT REGARDING FEDERALLY SPONSORED RESEARCH OR DEVELOPMENT

Not applicable.

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BACKGROUND OF THE INVENTION

20 This invention relates generally to oleochemical raw materials and
more particularly to a process for the enzymatic deacidification of natural
fats and oils.

Native fats and oils normally contain considerable quantities of fatty
acids as a result of enzymatic degradation reactions, their fatty acid content
being variable within wide limits according to provenance and previous
25 history, but almost always exceeding 3% by weight. These free fatty acids
have been found to be troublesome in the various processes used for
further processing of the triglycerides, more especially in the low-pressure
transesterification process for the production of fatty acid methyl esters.
Accordingly, it is normally not possible to avoid converting the fatty acids
30 into alkyl or glycerol esters by refining or preliminary esterification with
lower alcohols. However, this is very time- and energy-consuming,

especially since large excesses of alcohol have to be used and the necessary catalysts are difficult to remove.

Accordingly, the problem addressed by the invention was to provide a new process for the deacidification of fats and oils which would be free
5 from the disadvantages mentioned.

BRIEF SUMMARY OF THE INVENTION

Deacidified fats and/or oils are made by the process which comprises the steps of:

- 10 (a) reacting a technical triglyceride having an acid value of up to about 60 and an excess of a lower alcohol having from 1 to 4 carbon atoms and an effective amount of a lipase to form a pre-esterification product having an acid value of from about 0.5 to about 10,
- 15 (b) optionally removing water and unreacted alcohol from the pre-esterification product,
- (c) further reacting the pre-esterification product from step (a) or (b) with additional lower alcohol to form a post-esterification reaction product having an acid value of from about 0.1 to about 0.5.

20

BRIEF DESCRIPTION OF THE SEVERAL VIEWS OF THE DRAWING

Not Applicable.

25

DETAILED DESCRIPTION OF THE INVENTION

The present invention relates to a process for the production of deacidified fats and/or oils, in which

- 30 (a) technical triglycerides with acid values of up to 60 are treated with an excess of lower aliphatic alcohols in the presence of an effective amount of a lipase to form a pre-esterification product which still has an acid value of 0.5 to 10,

- (b) water and unreacted alcohol are optionally removed from the pre-esterification product,
- (c) the optionally dried pre-esterification product is subjected in the presence of more lower aliphatic alcohol to a post-esterification reaction in which the acid value of the starting materials is reduced to 0.1 to 0.5.

It has surprisingly been found that the acid values of even particularly acidic fats and oils can be reduced quickly, reliably and with minimal outlay on equipment to below 0.5 by the process according to the invention so that starting materials thus refined are eminently suitable for use as such, i.e. without further purification, in the low-pressure transesterification process described, for example, in German Patent application **DE-A1 3932514**.

Triglycerides

Basically, the choice of the fats and oils to be deacidified is not critical. The triglycerides normally used correspond to formula (I):



in which R^1CO , R^2CO and R^3CO independently of one another represent linear and/or branched, saturated and/or unsaturated acyl groups containing 6 to 24 and preferably 12 to 18 carbon atoms and 0 and/or 1 to 3 double bonds. From their production, the starting materials contain significant quantities of free fatty acids, so that they can have acid values of up to 60 and preferably in the range from 15 to 50. In consequence of the partial saponification, the triglycerides may also contain corresponding quantities of partial glycerides, i.e. mono- and diglycerides. The process

according to the invention may employ naturally occurring or synthetic triglycerides or a combination thereof. The preferred triglycerides are natural fats and oils such as, for example, palm oil, palm kernel oil, olive oil, olive kernel oil, sunflower oil from old and new plants, rapeseed oil from old and new plants, linseed oil, soybean oil, bovine tallow and fish oil. However, it is preferably applied to technical coconut oils which have acid values of from about 15 to about 60. If only mildly acidic fats and oils are used, the acid value can be raised to a maximum of about 60 and preferably about 20 by the addition of free fatty acids, for example refining fatty acids. In this way, the quantity of alkyl ester is optimized and the process made particularly economical.

Aliphatic alcohols

Basically, suitable lower aliphatic alcohols which are added to and mixed with the triglycerides during the pre- and the post-esterification steps are those containing 1 to 4 carbon atoms, i.e. for example ethanol, propanol, isopropyl alcohol and the isomeric butanols. However, the deacidified triglycerides are normally transesterified to the fatty acid methyl esters which are then hydrogenated to the fatty alcohols. Accordingly, the preferred alcohol is methanol. The quantities of alcohol which can be used in steps (a) and (c) is an excess amount so that, preferably, additions of from about 1 to about 10% by weight and preferably from about 3 to about 8% by weight, based on the triglycerides, in each of the two steps have proved successful.

Lipases

The use of lipases for the esterification of fatty acids with alcohols is well-known. From the wealth of prior art literature available on the subject, reference is made purely by way of example at this juncture to the two articles in *J. Am. Oil Chem. Soc.* **65**, 927 (1988) and *ibid.* **71**, 927 (1994).

The reduction of the acid value by pre-esterification with glycerol is the subject of Japanese patent application **JP Hei 04/183396**. The choice of the lipases used for the pre- and post-esterification steps is again not critical. Basically, any of the representatives known from the literature, such as for example *Candida cylindracea*, *Aspergillus niger* or *Pseudomonas fluorescens*, may be used. However, *Candida antarctica* has proved to be particularly effective. The quantity of lipases used is an effective amount which is any amount required to bring about the desired acid value. Typically, the amount of the lipase will be between about 0.5 and about 5% by weight of the triglyceride and is preferably from about 2 to about 4% by weight, based on the triglycerides, a substantially linear increase in the reaction rate being observed up to a quantity of about 4% by weight.

15 Pre- and post-esterification

The pre- and post-esterification steps may be carried out in known manner. The acidic fats and oils are mixed with the lipase and the lower alcohols, optionally with stirring. The pre-esterification temperature may be from about 10 to about 50°C but is preferably in the range from about 20 to about 40°C. Once the product has reached an acid value of from about 0.5 to about 10, which can readily be established by sampling, the water of reaction formed is removed in a preferred embodiment of the invention in order to enable the equilibrium to be shifted further to the product side in the post-esterification. At the same time, the unreacted alcohol is at least partly removed but may be returned to the reaction later after working up. The removal of water may be dispensed with providing the quantity of enzyme is sufficiently large, for example of the order of about 4% by weight. Under these conditions, pre- and post-esterification coincide. A second quantity of the alcohol is then added to the mixture optionally freed from water beforehand. The post-esterification is carried out under the

same conditions as the pre-esterification and is terminated when the required acid number has been reached. The small quantities of water formed in the post-esterification may remain in the end product together with the unreacted alcohol and the lipase because they do not affect the subsequent low-pressure transesterification.

The following examples are meant to illustrate but not to limit the invention.

- Example 1.** To crude coconut oil with an acid value of 8.2 were added 2% by weight - based on the triglyceride - of commercially available lipase of the *Candida antarctica* type (NOVOZYM® 435) and 5% by weight - again based on the triglyceride - of methanol, followed by stirring at 30°C. After 1 hour, an acid value of 1.05 had been reached. The water of reaction was then distilled off in vacuo together with the unreacted alcohol and another 5% by weight of methanol was added to the mixture remaining behind. The mixture was then stirred for another hour at 30°C and post-esterified at the same time, the acid value falling to 0.41.

- Example 2.** Example 1 was repeated using 4% by weight of lipase; the water of reaction was not removed. An acid value of 0.5 was reached after a reaction time of only 1 hour.

What is claimed is:

1. A process for the production of deacidified fats and/or oils comprising the steps of:
 - 5 (a) reacting a technical triglyceride having an acid value of up to about 60 and an excess of a lower alcohol having from 1 to 4 carbon atoms and an effective amount of a lipase to form a pre-esterification product having an acid value of from about 0.5 to about 10,
 - (b) optionally removing water and unreacted alcohol from the pre-esterification product,
 - 10 (c) further reacting the pre-esterification product from step (a) or (b) with additional lower alcohol to form a post-esterification reaction product having an acid value of from about 0.1 to about 0.5.
- 15 2. The process of claim 1 wherein the technical triglyceride is a compound of the formula (I):



wherein each of R^1CO , R^2CO and R^3CO is a linear and/or branched, saturated and/or unsaturated acyl group having from about 6 to about 24 carbon atoms and having up to 3 double bonds.

- 25 3. The process of claim 1 wherein the triglyceride is a synthetic triglyceride, a natural triglyceride or a combination thereof.
4. The process of claim 1 wherein the triglyceride is coconut oil having
- 30 an acid value of from about 15 to about 60.

5. The process of claim 1 wherein the acid value of the triglyceride is increased to a maximum acid value of about 60 by the addition of a fatty acid.
- 5 6. The process of claim 1 wherein the lower alcohol is methanol.
7. The process of claim 1 wherein the amount of the lower alcohol is from about 1 to about 10% by weight of the triglyceride.
- 10 8. The process of claim 1 wherein the lipase is *Candida antarctica*.
9. The process of claim 1 wherein the amount of the lipase is from about 0.5 to about 5% by weight of the triglyceride.
- 15 10. The process of claim 1 wherein steps (a) and (c) are each carried out at a temperature of from about 10 to about 50°C.

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ABSTRACT OF THE DISCLOSURE

Deacidified fats and/or oils are made by the process which comprises the steps of:

- 5 (a) reacting a technical triglyceride having an acid value of up to about 60 and a stoichiometric excess of a lower alcohol having from 1 to 4 carbon atoms and from about 0.5% to about 5% by weight of a lipase to form a pre-esterification product having an acid value of from about 0.5 to about 10,
- 10 (b) optionally removing water and unreacted alcohol from the pre-esterification product,
- (c) further reacting the pre-esterification product from step (a) or (b) with additional lower alcohol to form a post-esterification reaction product having an acid value of from about 0.1 to about 0.5.

Cognis Corporation, Patent Dept.
2500 Renaissance Blvd., Suite 200
Gulph Mills, PA 19406

JED/ras

Type a plus sign (+) inside this box ☐

0019/PTO
Rev. 6/95

U.S. Department of Commerce
Patent and Trademark Office

DECLARATION FOR UTILITY OR DESIGN PATENT APPLICATION

☒ Declaration Submitted with Initial Filing OR ☐ Declaration Submitted after Initial Filing

Attorney Docket
Number

C 2109 COGG

First Named
Inventor

BUETTGEN, Karl-Heinz

COMPLETE IF KNOWN

Application Number

Filing Date

Group Art Unit

Examiner Name

As a below named inventor, I hereby declare that:

My residence, post office address, and citizenship are as stated below next to my name.

I believe I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled:

PROCESS FOR THE PRODUCTION OF DEACIDIFIED TRIGLYCERIDES

(Title of the Invention)

the specification of which

☒ is attached hereto

OR

☐ was filed on (MM/DD/YYYY) as United States Application Number or PCT International

Application Number and was amended on (MM/DD/YYYY) (if applicable).

I hereby state that I have reviewed and understand the contents of the above identified specification, including the claims, as amended by any amendment specifically referred to above.

I acknowledge the duty to disclose information which is material to patentability as defined in Title 37 Code of Federal Regulations, § 1.56.

I hereby claim foreign priority benefits under Title 35, United States Code §119(a)-(d) or §365(b) of any foreign application(s) for patent or inventor's certificate, or §365(a) of any PCT International application which designated at least one country other than the United States of America, listed below and have also identified below, by checking the box, any foreign application for patent or inventor's certificate, or of any PCT International application having a filing date before that of the application on which priority is claimed.

Prior Foreign Application Number(s)	Country	Foreign Filing Date (MM/DD/YYYY)	Priority Not Claimed	Certified Copy Attached? YES NO
199 56 599 6	Germany	11/25/1999	<input type="checkbox"/>	<input checked="" type="checkbox"/> <input type="checkbox"/>
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			<input type="checkbox"/>	<input type="checkbox"/> <input type="checkbox"/>

☐ Additional foreign application numbers are listed on a supplemental priority sheet attached hereto:

I hereby claim the benefit under Title 35, United States Code §119(e) of any United States provisional application(s) listed below.

Application Number(s)	Filing Date (MM/DD/YYYY)	Additional provisional application numbers are listed on a supplemental priority sheet attached hereto.
		<input type="checkbox"/>

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DECLARATION

Page 2

I hereby claim the benefit under Title 35, United States Code § 120 of any United States application(s), or § 365(c) of any PCT international application designating the United States of America, listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in the prior United States or PCT international application in the manner provided by the first paragraph of Title 35, United States Code § 112, I acknowledge the duty to disclose information which is material to patentability as defined in Title 37, Code of Federal Regulations § 1.56 which became available before the filing date of the prior application and the national or PCT international filing date of this application.

U.S. Parent Application Number	PCT Parent Number	Parent Filing Date (MM/DD/YYYY)	Parent Patent Number (if applicable)

☐ Additional U.S. or PCT international application numbers are listed on a supplemental priority sheet attached hereto.

As a named inventor, I hereby appoint the following attorney(s) and/or agent(s) to prosecute this application and to transact all business in the Patent and Trademark Office connected therewith:

☐ Firm Name Customer Number OR ☒ List Attorney(s) and/or agent(s) name and registration number below:

Name	Registration Number	Name	Registration Number
John E. Drach	32,891	Aaron R. Ettelman	42,516
Steven J. Trzaska	36,296	Henry E. Millson, Jr.	18,980

☐ Additional attorney(s) and/or agent(s) named on a supplemental sheet attached hereto.

Please direct all correspondence to: ☒ Customer Number **23657** OR ☒ Fill in correspondence address below

Name	John E. Drach		
Address			
Address			
City	State	ZIP	
Country	Telephone	610-278-4925	Fax 610-278-6548

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

Name of Sole or First Inventor:		<input type="checkbox"/> A petition has been filed for this unsigned	
Given Name	Karl-Heinz	Middle Initial	
Family Name	Buettgen	Suffix e.g. Jr.	
Inventor's Signature	Date		
Residence: City	Kerpen	State	
	Country	Germany	Citizenship Germany
Post Office Address	Breite Strasse 67		
Post Office Address			
City	50170 Kerpen	State	
	Zip	Country	Germany
<input checked="" type="checkbox"/> Additional inventors are being named on supplemental sheet(s) attached hereto		Applicant Authority	

DECLARATION**ADDITIONAL INVENTOR(S)
Supplemental Sheet**

Name of Additional Joint Inventor, if any:



A petition has been filed for this unsigned inventor

Given
Name**Manfred**Middle
InitialFamily
Name**Lindemann**Suffix
e.g. Jr.Inventor's
Signature

Date

Residence:
City**Solingen**

State

Country

Germany

Citizenship

Germany

Post Office Address

Eipassstrasse 83

Post Office Address

City

42719 Solingen

State

Zip

Country

GermanyApplicant
Authority

Name of Additional Joint Inventor, if any:



A petition has been filed for this unsigned inventor

Given
Name**Daniela**Middle
InitialFamily
Name**Prinz**Suffix
e.g. Jr.Inventor's
Signature

Date

Residence:
City**Monheim**

State

Country

Germany

Citizenship

Germany

Post Office Address

Rasenstrasse 7

Post Office Address

City

40789 Monheim

State

Zip

Country

GermanyApplicant
Authority

Name of Additional Joint Inventor, if any:



A petition has been filed for this unsigned inventor

Given
NameMiddle
InitialFamily
NameSuffix
e.g. Jr.Inventor's
Signature

Date

Residence:
City

State

Country

Citizenship

Post Office Address

Post Office Address

City

State

Zip

Country

Applicant
Authority

Name of Additional Joint Inventor, if any:



A petition has been filed for this unsigned inventor

Given
NameMiddle
InitialFamily
NameSuffix
e.g. Jr.Inventor's
Signature

Date

Residence:
City

State

Country

Citizenship

Post Office Address

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City

State

Zip

Country

Applicant
Authority☐ Additional inventors are being named on supplemental sheet(s) attached hereto

09717894-11210